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APTD-1109

PB 209 269

FINAL REPORT

IMPROVED CHEMICAL METHODS FOR SAMPLING AND ANALYSIS OF GASEOUS POLLUTANTS FROM THE COMBUSTION OF FOSSIL FUELS

VOLUME III
CARBON MONOXIDE

Contract No. CPA 22-69-95

JULY 1971

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Prepared for

ENVIRONMENTAL PROTECTION AGENCY
Cincinnati, Ohio

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12

BIBLIOGRAPHIC DATA SHEET	1. Report No. APTD-1109	2.	3. Recipient's Accession No.

4. Title and Subtitle
Improved Chemical Methods for Sampling and Analysis of Gaseous Pollutants from the Combustion of Fossil Fuels Volume III Carbon Monoxide

5. Report Date
July 1971

6.

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10. Project/Task/Work Unit No.

11. Contract/Grant No.
CPA 22-69-95

12. Sponsoring Organization Name and Address
ENVIRONMENTAL PROTECTION AGENCY, Office of Air Programs
Division of Control Systems, Process Measurements Section
Cincinnati, Ohio

13. Type of Report & Period Covered
Final

14.

15. Supplementary Notes

16. Abstracts

State-of-the-art manual methods are examined for sampling and analysis of carbon monoxide in stationary fossil fuel combustion sources for the concentration range 10-1000 ppm.

17. Key Words and Document Analysis. 17a. Descriptors

Air pollution
Carbon monoxide
Combustion products
Chemical analysis
Gas sampling

17b. Identifiers/Open-Ended Terms

Sources

17c. COSATI Field/Group 13B

18. Availability Statement	19. Security Class (This Report)	21. No. of Pages
Unlimited	UNCLASSIFIED	75
	20. Security Class (This Page) UNCLASSIFIED	22. Price

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by
J. N. Driscoll
and
A. W. Berger

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FOREWORD

The compilation of information contained in this publication was performed pursuant to Contract No. CPA 22-69-95, Air Pollution Control Office, Environmental Protection Agency.

The information was compiled by Walden Research Corporation and their subcontractor, Arthur D. Little, during the period June 12, 1969 to September 11, 1970.

Volume I of this report examines state-of-the-art manual methods for sampling and analysis of sulfur oxides. The determination of sulfur dioxide (10-3000 ppm) and sulfur trioxide (5-300 ppm) is reviewed for stationary fossil fuel combustion sources and for control equipment which may be installed to reduce the emissions from such sources.

Volume II reviews methods for the determination of oxides of nitrogen in stationary fossil fuel combustion sources for the concentration range 5-2000 ppm for nitric oxide (NO), nitrogen dioxide (NO_2), and total oxides of nitrogen (NO + NO_2 or NO_x).

Volume III of this report examines state-of-the-art manual methods for sampling and analysis of carbon monoxide in stationary fossil fuel combustion sources for the concentration range 10-1000 ppm.

The major pollutants of interest in this study are sulfur and nitrogen oxides. The report subsequently includes a literature search, review of methodology and laboratory investigation on the most promising methods. Carbon monoxide, the minor pollutant, was limited to a literature search and review of methodology.

This report has been reviewed and approved.

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1.0 Introduction

This is Volume III of three volumes of the Final Report on "Improved Chemical Methods for Sampling and Analysis of Gaseous Pollutants from the Combustion of Fossil Fuels (in Stationary Sources)." The three volumes are divided by subject as follows:

I Sulfur Oxides
II Nitrogen Oxides
III Carbon Monoxide

This report was prepared under Contract No. CPA 22-69-95 covering the period June 12, 1969 to September 11, 1970. The principal objectives of this program were: (1) to review the state-of-the-art for manual (wet chemical) methods for the determination of emissions of specified gaseous pollutants from (indirect-fired) stationary fossil fuel sources, (2) to select and develop improved procedures for high precision emissions determinations, and (3) to select and develop simplified procedures where high precision determinations were judged to be complex (i.e., required a highly trained research and development group).

General principles of flue gas sampling have been discussed in Volume I of this report. However, problems specific to sampling for the carbon monoxide are included. The combustion effluent environment is treated in similar fashion.

In the following subsections, we consider method development priorities based upon national CO emissions and summarize the literature search of the state-of-the-art in manual determination of CO in combustion effluents.

1.1 National CO Emissions and Method Application Priorities

In contrast to both SO_x and NO_x emissions patterns, only a very small fraction of total CO emissions (~1%, Ref. 257) are attributable to stationary fossil fuel combustion sources. All such CO emissions (0.9×10^6 ton/yr, 1966, Ref. 257) are assigned by NAPCA to coal-fired sources.

CO emissions from grate-fired units (see Table 2-4 following) are generally larger than from suspension-fired units and the emissions from small units are larger (lbs/10^6 BTU basis) than those from large units. (See Section 2, following).

Although it is thus relatively simple to isolate the major sources of CO emissions, CO analyses will often be conducted for diagnostic purposes, i.e., to determine combustion control efficiency or in process loops of control equipment.

In summary, CO method application priorities emphasize small grate-fired units which appear to be the major sources, but include capability for large equipment at relatively low CO levels.

In the following section, we describe the combustion effluent environment.

2.0 The Combustion Effluent Environment

2.1 Introduction

To aid in the selection of manual chemical methods for the sampling and analysis of carbon monoxide (CO) from stationary combustion sources a description of the effluent environment is needed. The sources considered include: steam generators, water heaters, process heaters, space heaters and stationary internal combustion engines; excluded are combustion devices in which process gas emissions are combined with the combustion gases (e.g., lime kilns). In general, measurements of carbon monoxide emissions will be at stack conditions or at the inlet and outlet of effluent control equipment.

The major results and conclusion from the Phase I Report,

Part A, in which the full combustion effluent environment was discussed, are reviewed here. The areas pertinent to analysis for CO are discussed in further detail.

2.2 Summary of Results from Volume I

Combustion equipment was arbitrarily divided into three size groups as shown in Table 2-1. Each of these three groups was then subdivided by fuel type, namely, coal, oil, and gas. No attempt has been made to identify the environment for specific furnaces with particular operating conditions and practices. Rather, the goal has been to report ranges and average emissions and concentrations from the literature. These are shown in Table 2-2. The average requirements for sampling and analysis methods are based upon these environments.

2.3 Carbon Monoxide Emission

The choice of analytical equipment and technique will be dictated primarily by the concentration level of carbon monoxide, temperature, and the presence of other chemical species. Concentration fluctuations are also of importance. The range of concentrations and the range of emissions of CO for equipment and fuel categories previously used are shown in Table 2-3. In general, small, oil and coal burning combustion devices yield higher concentrations of CO than large units regardless of fuel type. These differences result primarily from the degree and type of controls used and needed. On large units the controls are adequate and maintenance of these controls is essential since small losses in combustion efficiency represent large costs. On small oil and coal burning units the same types of control and maintenance are needed for good control but the economic incentives are

TABLE 2-1

Size Categories for Stationary Combustion Sources

	LARGE	INTERMEDIATE	SMALL
BTU per hour	$\geq 5 \times 10^8$	3×10^5-5×10^8	$< 3 \times 10^5$
Boiler Horsepower	≥15,000	10-15,000	< 10
Pounds of Steam per hour	≥500,000	350-500,000	< 330
Megawatts	≥50	< 50MW	Not Applicable

TABLE 2-2

EMISSION PARAMETERS FOR FOSSIL FUEL COMBUSTION[a] (pounds/10^6 BTU)

	GAS[b]			OIL[c]			COAL[d]		
	Large	Intermed.	Small	Large (Resid.)	Intermed. (Resid./Dist.)	Small (Dist.)	Large	Intermed.	Small
Velocity ft/min	2000-3000			2000-3000			2000-3000		
Moisture	18% @ 0% XS Air 15% @ 20% XS Air			10% @ 0% XS Air 9% @ 20% XS Air			6% (Nominal)		
Excess Air %	15	15-75	75	20	20-75	75	25	25-75	75-100
Stack Temp. °F (of Breeching)	275-400	400-750	750-900	300-400	400-750	750-900	300-400	400-750	750-900
CO	Neg.	$4x10^{-4}$	$4x10^{-4}$	$3x10^{-4}$	0.01/0.01	.01	0.02	0.1-2	2
SO_2	$4x10^{-4}$	$4x10^{-4}$	$4x10^{-4}$	1.65	1.65/0.31	0.31	3.5	3.5	3.5
SO_3				0.03	$0.016/3x10^{-3}$	$3x10^{-3}$			
NO	0.35	0.2	0.1	0.68	0.47/0.51	.08	0.8	0.8-0.3	0.3
NO_2									
Particulate	0.014	0.016	0.017	0.07	0.15/0.10	0.06	0.46 (90% cont.)	0.53 (65% cont.)	0.8 (uncont.)
Hydrocarbons	Neg.	Neg.	Neg.	0.022	0.01/0.01	.02	$8x10^{-3}$	0.04-0.4	0.4
Transients	10% of Capacity Per Min.			10% of Capacity Per Min.			5% of Capacity Per Min.		

a. Details in Appendix 1, Final Report, Vol. I

b. 1100 BTU/SCF

c. $S_{dist.}$ 0.39%, 142,000 BTU/Gal
$S_{resid.}$ 1.6%, 152,000 BTU/Gal

d. S = 2.5%, 8% Ash, 13,000 BTU/#

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TABLE 2-3

Range of Concentration and Emission of Carbon Monoxide from Combustion Devices (Possible High Values of Concentration Given in Parenthesis)

Type of Fuel	Concentration Range, ppm	Emissions Range, #/10^6 BTU
Coal		
Large	0-110	0.005 - 0.44
Medium	0-500	<0.1 - 2.0
Small	0-2000 (10,000)	1.1 - 3.5
Oil		
Large	<1 ppm	$3X10^{-4}$
Medium	0-120 (1100)	0 - 0.1
Small	0-1500(10,000)	0 - 1.0
Gas		
Large	negligible	negligible
Medium	negligible	$4X10^{-4}$
Small	negligible	$4X10^{-4}$

smaller. Gas firing presents a somewhat easier control and maintenance problem and is therefore, often used in small and medium sized units where control of the emissions is a primary objective.

The reported range of concentration of CO is very large, from essentially zero to 10,000 ppm. The upper range extends to 30,000 ppm if stationary internal combustion engines are included. The range of carbon monoxide concentration even for a given fuel type and unit size is still large (0-1500 ppm) for small sized, oil burning equipment. This extreme range probably represents lack of adequate control or maintenance on some units.

2.4 Control of Carbon Monoxide Emissions

With proper control and maintenance and with normal amounts of excess air, stack concentrations of CO should be very low. Essentially all combustion devices (excluding stationary internal combustion engines) are designed to achieve complete combustion. For those units in which particulate burnout is not rate limiting, the burning of CO, which is formed rapidly from hydrocarbons, is limiting. A typical burning law (258) for CO is:

$$\frac{-d(CO)}{dt} = 1.8 \times 10^{10} e^{-25,000/RT} \left(\frac{P}{RT}\right)^2 (f_{CO})(f_{H_2O})^{1/2}(f_{O_2})^{1/2} \qquad (1)$$

where the rates are in gr. moles/liter/sec., f is the mole fraction and P,R, and T represent pressure, the gas constant, and temperature, respectively. Most combustion devices are designed on the basis of carbon monoxide combustion. Clearly, high, local oxygen concentrations and tempera-

tures are important in maintaining high rates. Secondary considerations, which become important for very low concentrations, are equilibrium levels of CO and the rates of cooling of the combustion gases. In most cases, low levels of CO can be achieved by considering only the forward reaction rate (Equation 1) and allowing sufficient residence time.

High concentration levels of CO in combustion gases are likely from fixed-bed coal burning units, particularly small units without automatic controls. Emissions from various grate-fired units are compared in Table 2-4.

TABLE 2-4

Emissions from Grate-Fired Units (259)

Type of Unit	Size (10^6 BTU/hr)	CO Emission #/10^6 BTU
Chain Grate	147	0.51
Spreader Stoker	59.2	< 0.1
Underfed Stoker	4.4	0.16
Underfed Stoker	3.0	0.14
Underfed Stoker	0.066	1.1
Hand-fired Stoker	0.155	3.5

The carbon monoxide results from reduction of carbon dioxide in the presence of the hot carbon fuel in local excess.

Generally, the CO is burned to CO_2 with overfire air. The trend in the design of new equipment is toward suspension firing although stokers are still widely used (most small units are stokers). The coal burned in stokers is about 25% of the total coal consumption (260).

The concentration of carbon monoxide from internal combustion engines can reach high levels (30,000 ppm with spark-ignition). However, such engines represent only a small fraction of total stationary combustion and power CO emission. Many of the stationary engines are diesel, with lower CO emission levels. Recent trends in engine operation and control have lowered the average levels of CO significantly. It is unlikely that CO levels below 100-150 ppm will be achieved without major modifications to engines, however.

2.5 Chemical and Physical Interferences

Carbon monoxide is low boiling and not easily absorbed or adsorbed. (See Section 3.0). Thus, condensation or loss onto surfaces or into liquids is unlikely.

Many of the chemical components in the combustion gases, e.g., SO_2 and CO_2, can interfere with the analysis for carbon monoxide. These compounds are generally easily removed by absorption prior to analysis for CO, since CO is rather inert. Problems are associated with quantitative separation, however, as a result of the large concentration of interfering species which often exist. These interferences are discussed in detail for each specific method of analysis.

In the following section we briefly summarize CO chemistry relevant to flue gas analysis.

3.0 Chemistry of Carbon Monoxide in Flue Gas

3.1 Physical Properties

Carbon monoxide is a colorless, odorless, tasteless gas, slightly lighter than air (28:29). It is only slightly soluble in water with 100 volumes of water dissolving 3 volumes of the gas at 0°C. Liquid carbon monoxide boils at -192°C.

3.2 Physiological Action

The A.S.A. in cooperation with the U.S. Public Health Service sets the limit of carbon monoxide concentration for eight hours daily exposure at 100 parts carbon monoxide per million parts of air. The action of carbon monoxide as a poison results from the formation of a stable complex with hemoglobin and consequent reduction of the oxygen carrying capacity of the blood.

3.3 Absorption

Carbon monoxide is absorbed very slighly by charcoal at ambient temperatures. Gas masks designed for operation in carbon monoxide containing atmospheres usually contain a mixture of metallic oxides such as Ag_2O, Co_2O_3, MnO_2 and CuO employed to catalyze the oxidation of carbon monoxide to the dioxide at ordinary temperatures.

3.4 Chemical Reactions

Carbon monoxide characteristically reacts with various metals to form carbonyls. For common materials of construction, the most significant product species are: $Fe(CO)_5$, a yellow substance which melts at -20°C and boils at 103°C, and nickel carbonyl, $Ni(CO)_4$, which is colorless, melts at -23°C and boils at 43°C. Carbon monoxide is absorbed by acidic and

ammonical solutions of copper (I) chloride with the formation of addition compounds of the formula $CuCl \cdot CO \cdot 2H_2O$ and $CuCl \cdot CO \cdot NH_3$. These reactions provide the basis of the Orsat techniques for the determination of carbon monoxide concentration (261). Carbon monoxide may be oxidized to CO_2 by powerful oxidants at flue gas temperatures (262). The reaction with iodine pentoxide, is the basis for one method of analysis at low concentration.

$$I_2O_5 + 5CO \rightarrow 5CO_2 + I_2$$

3.5 The CO_2, C, CO Equilibrium

The equilibrium, $CO_2 + C = CO$, has considerable interest and importance. In the absence of a catalyst equilibrium is established slowly, but metals and metal oxides are often effective catalysts. In their presence, carbon may be deposited from carbon monoxide at quite low temperatures.

At equilibrium at a total pressure of 1 atmosphere, the amounts of carbon monoxide present are: 450°C, 2%; 600°C, 23%; 800°C, 94%; 1000°C, 99.3% (263).

The equilibrium has been studied by radioactive tracer techniques (264). The most probable reaction sequence is that in which the primary reaction is the rapid reduction of the dioxide to the monoxide; the oxygen removed remains absorbed on carbon (formulated below as C_nO):

$$CO_2 + C_n = CO + C_nO$$

This is followed by a much slower reaction in which the absorbed oxygen is desorbed as carbon monoxide: $C_nO \rightarrow CO + C_n$.

3.6 Conclusions

Carbon monoxide behaves as a permanent gas in most respects. Its relative inertness leads to few complications in detection and measurement. Under conditions existing in flue gases its ability to form carbonyls will not be important. The negligible rate of low temperature oxidation and small absorptivity make analytical techniques reliable, in the absence of interferences from other species.

4.0 Methods for the Determination of CO

4.1 Introduction

Manual methods for the analysis of carbon monoxide depend on one of the following reactions:

reduction of a metal

$$CO + MX_2 + H_2O \rightarrow M + CO_2 + 2HX$$

oxidation

$$CO \xrightarrow[\text{Catalyst}]{O_2} CO_2$$

complexation

$$CO + MX + xH_2O \rightarrow CO \cdot MX \cdot xH_2O$$

The "reduction" methods are attractive because of their simplicity and freedom from interferences; however, the long reaction time (for the reduction process) and low solubility of CO in aqueous solutions restrict these methods to grab sampling techniques.

The oxidation methods appear promising since a long (integrated) sampling time is preferred to eliminate transients occurring during flue gas sampling. The major difficulty is that CO_2 and other acidic flue gas compounds have to be quantitatively scrubbed prior to oxidation of CO.

The complexation methods have been used in power plants when combustion is poor and CO concentrations are, therefore, high (200-2000 ppm). Gas volumetric apparatus, such as the Haldane and Orsat, depend on selective scrubbing (absorption) of CO and measurement of the difference in volume absorbed in the cuprous scrubbing solution. The basic limitation of these volumetric techniques is the measurement of very small differential volumes. Since the carbon monoxide concentration in well-controlled combustion sources is < 100 ppm, absorption (complexation) methods lack adequate sensitivity. The solubility of CO in aqueous cuprous solutions has been found (265) to be proportional to the concentration of dissolved Cu+. (35-125 g/l). The solubility of absorbed CO approaches a maximum (with increasing pressure) of 1 mole of CO per mole of Cu+ in solution, or a maximum of 2 moles of CO per liter (56 g/l, compare Table 4-1). The maximum solubility will decrease as the partial pressure of CO is reduced. However, this approach may be feasible for collection of CO at low concentrations if an analytical technique other than volumetric readout is used (e.g., colorimetric). Other complexation techniques (blood methods) utilize the CO-hemoglobin complex for scrubbing. Complexation methods are of limited use for CO in fossil fuel effluents, and are included under miscellaneous methods. Thus, we have divided CO methodology into three sections:

a) "reduction" methods

b) oxidation methods

c) miscellaneous

Only the reduction and oxidation methods are considered in detail. The miscellaneous methods which include complexation techniques are included for completeness, only, but do not appear promising as used for the determination of CO (at low levels) in flue gases.

4.2 Reduction Methods

Since the slow reduction of the metallic halides and the low solubility of CO in aqueous solutions limits the sampling technique to grab sampling, we discuss grab sampling techniques in some detail in the following section.

4.2.1 Sampling

Gases which have a high solubility in aqueous solutions can be collected by absorption in a liquid since this is a prerequisite for efficient scrubbing. This technique is used for collection of the sulfur oxides which have a very high solubility in aqueous solutions. The solubilities of a number of common gases encountered in flue gas sampling are given in Table 4-1.

TABLE 4-1

Solubilities of Common Gases in Water at 20°C (266)

Gas	Solubility, g/l of water
Nitrogen	0.010
Carbon Monoxide	0.028
Oxygen	0.044
Nitric Oxide	0.063
Carbon Dioxide	1.74
Sulfur Dioxide	117

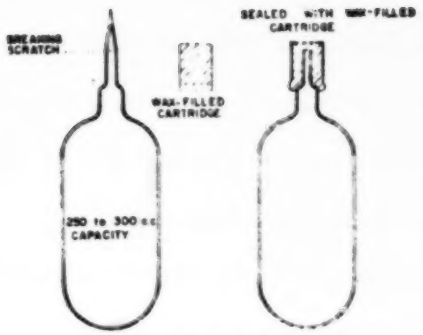
The low solubility of CO in aqueous solutions, therefore eliminates absorption in aqueous scrubbing solutions as a collection technique for CO. Although, the solubility of CO in many organic solvents (265) is higher than in aqueous solutions (e.g., 0.2 g/l in ethanol), it is still too low to be of utility for effective scrubbing of CO. Techniques used for collection of CO for, say, analysis by reduction methods are therefore restricted to grab samplers which utilize mechanical retention followed by absorption (Fig. 4-1, 4-2). In grab sampling techniques for NO_x (Phase I Report, Part B) the precision (coefficient of variation) of the PDS method is better than 5% at high NO_x concentrations (>500 ppm), but at concentrations of 100 ppm, the precision becomes poor (CV = ±10%). Since CO concentrations are not expected to exceed a maximum of ∿100 ppm for large units, a modified technique is needed for high precision results. For example, the collection of an integrated flue gas sample (by traverse) in a plastic bag followed by grab sampling for CO from the bag averages short term variations in concentration and should markedly improve precision.

4.2.2 Analytical Methods for CO by Reduction

.1 Reduction of Palladous Salts

a) Description of the Method (13,57,60,75,85,88,101, 125,151,191,192,223)

The gas sample containing CO is introduced into a 50-100 ml sample tube containing a few ml of a $PdCl_2$ solution. CO is oxidized according to the reaction, $PdCl_2 + CO + H_2O \rightarrow Pd + CO_2 + 2\ HCl$. Reaction times of 1 to 24 hours have been employed. The amount of $PdCl_2$ reduced has been determined by a variety of methods, including filtration of



Evacuated Sample Container.

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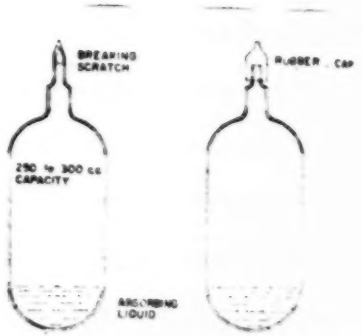
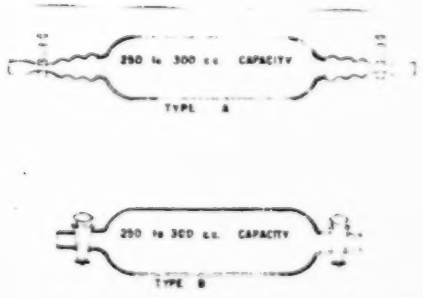
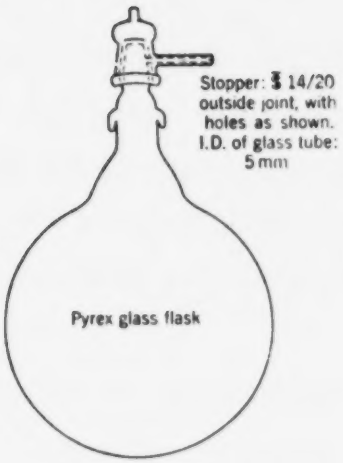


Evacuated Sample Container for Collection of Sample in an Absorbent.



Gas Sample Tubes for Collection of Sample by Displacement.

Figure 4-1 Types of grab samplers (partial)



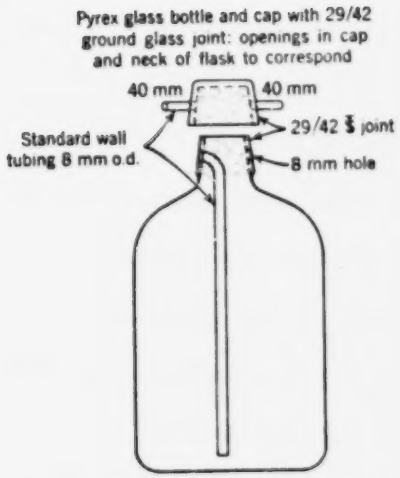

Pyrex glass bottle and cap with 29/42 ground glass joint: openings in cap and neck of flask to correspond
40 mm
40 mm
29/42 ⅄ joint
Standard wall tubing 8 mm o.d.
8 mm hole

Figure 4-2

Additional Grab Samplers (267)

the free Pd and subsequent weighing (125), reacting Pd with an excess of Br_2 and titrating the excess Br_2 (151), and photometric measurement of the Pd sol (88).

Another approach is the determination of the unreacted $PdCl_2$. After removing free Pd, the unreacted $PdCl_2$ may be converted to $PdI_4^=$ and measured spectrophotometrically at 406-410 nm (75,101,223). Phosphomolybdic acid reacts with $PdCl_2$ to form a blue heteropoly acid (molybdenum blue) which is determined by a filter photometer in the 635-720 nm region (85).

Bis(o-phenanthroline) forms a red-violet complex upon reaction with CO and $PdCl_2$; the absorbance is measured at 500 nm (60).

b) Areas of Application

The method has been used for analyses of air samples in mines (85), and for CO released from blood (13,75,162).

c) Applicable Concentration Range

A range of 0-4000 ppm is claimed for photometry of Pd sol. One measurement at 25 ppm agreed well with I_2O_5, but no real indication of lower limit was given (88).

Molybdenum blue measurement gives a range of 10-600 ppm to 5000 ppm by diluting the gas sample (85).

The $PdCl_2$-bis(o-phenanthroline) complex yields a range of 5-200 ppm. This can be extended upward by sample dilution.

d) Precision and Accuracy

Determination of CO with $PdI_4^=$ gives 99.6% recovery with $\pm$2.2% precision (223).

The precision of the phosphomolybdic acid-$PdCl_2$ method is 5-10% with an accuracy of about 10% at 600 ppm (85).

The precision of the bis(o-phenanthroline) method is given as 5-10% (60).

e) Interferences

These methods are generally not specific for CO. Hydrogen and unsaturated hydrocarbons are the primary interferences (88,125). Interferences were studied in detail for the phosphomolybdate method (85).; 1 ml of the following gases produced the following effects:

Compound	Equivalent to ml CO
Ethylene	0.7
Hydrogen Sulfide	0.7
Acetylene	0.5
Hydrogen	0.03
Gasoline	0.02-0.04
Sulfur Dioxide	0.0018
Benzene	0.000025

The $PdCl_2$-bis(o-phenanthroline) method was reported to be relatively free of interferences. Only H_2, at levels greater than 15%, H_2S and C_2H_2 were reported to have an effect (60). An interference due to slow oxidation of free Pd by O_2 was reported in one paper (223). At the optimum two-hour reaction time, 99.6% recovery was obtained; 22 hours later the recovery was only 91% due to air oxidation.

f) Comments

The $PdCl_2$-bis(o-phenanthroline) method is

attractive because of the short (25 min.) reaction time. However, the methodology and interferences (common to combustion flue gases) should be studied in more detail. The $PdCl_2$-phosphomolybdic variation (85) and Allen and Roots' $PdCl_2$ method (225) have been studied thoroughly and appear quite good.

g) Conclusions

Since Pd^{+2} reduction requires long reaction times under controlled conditions, a grab sample is required. Spectrophotometry of $PdI_4^=$ or molybdenum blue provides candidate simplified methods; $PdCl_2$-bis(o-phenanthroline) appears even more desirable.

.2 Reduction of Silver p-Sulfaminobenzoate

a) Description of the Method (1,10,53,87,94,157, 166,200,229,237)

Carbon monoxide in air reacts with an alkaline solution of the silver salt of p-sulfaminobenzoic acid to produce colloidal silver. The reagent consists of 0.1 M p-sulfaminobenzoic acid, 0.1 M $AgNO_3$, and 1 M NaOH mixed in a volume ratio of 2:2:1. In typical procedure 10 ml reagent is introduced into a 125 ml flask, and pumped until the reagent begins to bubble. A 50 ml sample of the gas to be analyzed is injected via syringe and the mixture is shaken for 2 hours. The absorbance of the solution is read at 425 nm for 0-400 ppm CO or at 600 nm for 0-1800 ppm CO. Calibration curves are reported to be linear for these ranges (10).

b) Areas of Application

This method has been applied primarily to analysis of CO in ambient air or blood. One application to incinerator

effluents has been reported (10).

c) Applicable Concentration Range

For ambient air samples, the range was from 10-2000 ppm (87,200). For incinerator effluents the range was 5-1800 ppm (10).

d) Precision and Accuracy

The reported precision is about ±5% (10,53, 94,166). A recovery of 95% has been reported (10).

e) Interferences

In the incinerator work (10), only acetylene and formaldehyde were found to interfere. These interferences at up to 1200 ppm and 100 ppm respectively were effectively removed with a bed of acidic $HgSO_4$ on silica. Hydrogen is slowly oxidized by the reagent if long reaction times are used (157). With a reaction time of 2 hours, however, up to 0.1% H_2 has no effect (10). Ammonia and H_2S are reported to interfere to some extent (157).

f) Comments

The absorbance of the silver sol decreases rapidly at longer wavelength and this is the basis for the dual concentration range method (10). The relative effect of acetylene interference is from four to ten times less at 425 nm than at 600 nm (10). A similar effect for H_2 vs. CO is evident in Ciuhandu's data (157); his spectra seem to indicate that the absorbance of the silver sol at wavelengths below 400 nm should be investigated for even more selectivity for CO in the presence of interferences. The reaction of the silver salt with CO is slow but with good control of reaction conditions, and use of a calibration curve, good

performance has been reported.

g) Conclusions

The slow reaction rate necessitates use of a grab sample. The reported freedom from interferences, and simplicity, make this method attractive.

4.3 Oxidation Methods

4.3.1 Sampling

Since these methods involve oxidation of CO to CO_2, release of iodine, etc., the products formed can be continuously collected in an absorbing solution due to their high solubility. Thus, the precision of these methods should be superior to grab sampling techniques because of the long integration times and the ability to collect a larger sample.

The major problem with these techniques is that CO_2 and oxidizing or reducing gases have to be quantitatively scrubbed to eliminate interferences. If we consider an oxidation method such as hopcalite, which involves oxidation of CO to CO_2, then determination of the CO_2 concentration, CO_2 must be reduced from 10^5 ppm to 1 ppm in order to achieve $\pm$10% accuracy for CO @ 10 ppm. The scrubbing efficiency of $Ba(OH)_2$ solution for CO_2 is only 60-80% (267), clearly not acceptable. A number of investigators have attempted to increase the collection efficiency of CO_2 in alkaline solution (243,244,253) by addition of monoethanol amine or carbonic anhydrase enzyme (243), or pyridine plus monoethanol amine (244). Their results show considerable increase in the collection efficiency but should be verified.

Solid sorbents such as ascarite may be considerably more effective for removal of CO_2. In view of the advantages and simplicity of the method, this is an attractive approach, although much laboratory work on CO_2 collection efficiencies has to be done before feasibility can be determined.

4.3.2 Analytical Methodology for Oxidation Methods

.1 Catalytic Oxidation of CO

a) Description of the Method

Gaseous samples are passed over catalysts such as heated CuO, CuO on silica (194), hopcalite (99,118), or silver oxide (189). The catalyst effluent is passed through 0.005 N $Ba(OH)_2$ which absorbs the CO_2 formed. The carbon dioxide is commonly titrated with oxalic acid and phenolphthalein indicator (28,99,194). A colorimetric procedure also has been based on this reaction (12). Another approach is the collection of CO_2 in $Ba(OH)_2$ solution followed by a conductivity readout (44,52). Other methods for CO_2 determination, e.g., titrimetry, conductivity, amperometry, etc. are given in references 242-256.

b) Areas of Application

Flue gas and ambient air measurements.

c) Applicable Concentration Range

The typical range is 10-5000 ppm, however, the photometric titration has been used over the range of 1-10 ppm (50).

d) Precision and Accuracy

Accuracy 10-20 ppm, precision ±3-5%.

e) Interferences

Most hydrocarbons except methane are oxidized to some extent and will interfere. Hydrocarbon interference is removed with

activated charcoal. The interference can be minimized when using Hopcalite by operating at room temperature (99). Ascarite-soda lime is used to remove CO_2 and other acid gases which would interfere.

f) Comments

It is feasible to take the apparatus (purification train, catalyst, and CO_2 absorber) into the field. The technique is adaptable to continuous sampling. For field use it is desirable to operate with the catalyst at ambient temperature. Hopcalite (99) and silver oxide (118,189) catalysts have different room temperature activities for the oxidation of trace hydrocarbons. The tendency for the unheated catalyst to retain some CO_2 has been noted (99,189) but not evaluated in detail. The major problem to be solved is the quantitative removal of CO_2.

g) Conclusions

Catalytic oxidation and titration of CO_2 collected in basic solution appears to be an attractive approach for sampling CO in the field, provided CO_2 can be quantitatively scrubbed prior to CO oxidation.

.2 Reaction with Iodine Pentoxide

a) Description of Method (8,15,52,61,73,89,112, 224,138,140,144,150,152,164,165,170,199,202,241)

The sample is passed at a rate of about 4ℓ/min through a purifying and drying train and then through a tube containing iodine pentoxide maintained at a constant temperature in the range 120 - 150°C. Carbon monoxide is oxidized to CO_2 in the reaction, $5CO + I_2O_5 \rightarrow I_2 + 5CO_2$

with quantitative release of free I_2 vapor.

In what is termed the classical method, the free I_2 is absorbed into 2-5 ml of 2-10% KI and titrated with 0.002 N sodium thiosulfate using starch indicator. For determining very small amounts of CO (< 30μg), Glover (61) trapped the I_2 on silica gel which was then transferred directly into 0.07 ml of KI. The I_2 was titrated with N/560 $Na_2S_2O_3$ using a microburette. For CO in the range of 1-100 ppm, the I_2 was trapped in 2.5% KI solution and measured spectrophotometrically at 289 nm (15).

b) Areas of Application

Ambient air (14,65,97,150,152,165,199,202)

Air in mines and tunnels (61,73,89,140,214)

Gaseous fuels (15,66,158,199)

Exhaust gases from engines and flames (137, 138,170)

c) Applicable Concentration Range

The concentration range is from 10 ppm to 0.5% by iodometry using sample volumes ranging from 5 to 1ℓ (241). Higher concentrations can be measured if steps are taken to avoid losses during I_2 or CO_2 absorption (114,140,164). The minimum detectability is usually stated to be about 1 ppm (61,73).

d) Precision and Accuracy

The precision of the iodometric titration was found to be ±1 ppm at the lower concentrations and ±10 ppm at 0.5% (241). Other studies claimed a precision of ±4% relative over the range 4-500 ppm (73) and ±5% relative from 10-1000 ppm (138).

With a properly conditioned I_2O_5 tube, CO conversions of from 97 to 99% have been reported (8).

e) Interferences (8,15,89,112,199)

The oxidation is not specific for CO and most oxidizable compounds with the exception of methane will interfere if present at a higher concentration than CO. Unsaturated hydrocarbons such as C_2H_2 and C_2H_4 are particularly troublesome because of the difficulty of their removal (112). Saturated hydrocarbons are also oxidized, but the series ethane through butane react with I_2O_5 only to the extent of 2-3% (8). Sulfur and nitrogen oxides, as well as H_2S and HCl, interfere in the determination by ruining the I_2O_5 reagent.

In the iodine finish, 1 ml H_2 produces a result equivalent to 0.003 ml of CO (8). If the CO_2 finish is used, the presence of H_2 can be tolerated, but CO_2 and other acid gases must be removed ahead of the I_2O_5 tube. Water degrades the I_2O_5 performance and must be removed.

Unsaturates are removed by a chromic-sulfuric acid solution scrubbing solution. More recently a solid reagent made by depositing an acidic solution of mercuric sulfate saturated with magnesium sulfate onto silica gel has been employed for removal of unsaturates (8). Silica impregnated with chromic acid is often used in conjunction with the $HgSO_4$ bed.

Drying has been accomplished with P_2O_5 (241) or silica gel activated at 300°C (197). Carbon dioxide can be removed with a bed of ascarite and/or KOH (52). Activated charcoal is used to remove heavier, saturated hydrocarbons.

f) Comments

The most difficult step in this method appears to be creating and maintaining a stable, reactive I_2O_5 bed. It is reportedly necessary to have about 0.2% water remaining in the I_2O_5. Conditioning prior to use ranges from 4 hours (241) to 2 days (15) at 225-230°C. Once conditioned, the I_2O_5 tube cannot be cooled from its operating temperature of $\sim$ 140°C without reconditioning.

Minimizing the effects of interfering species may require trial-and-error compromises. Oxidation of H_2 and saturated hydrocarbons can be reduced by operating the I_2O_5 tube at a lower temperature; risk of incomplete CO oxidation is then increased. The quantities of materials in the purifying train ($HgSO_4$-$MgSO_4$, silica, charcoal) can be increased for more efficient operation. This can only be done at the increased risk of retaining significant amounts of CO on the charcoal and silica and of oxidizing some CO on the $HgSO_4$.

In the earlier work employing liquid scrubbers, from 2-4 hours was required to perform the oxidation of a sample. Newer approaches utilize solid purification beds and reduce the time required to sweep the sample into and the I_2 out of the system; ten minutes is sufficient for a 250 ml sample (15). In fact, sweeping for too long a period can lead to errors as hydrocarbons initially trapped on the charcoal begin to elute into the I_2O_5. If hydrocarbons are present, a new charcoal trap may be required for each run, or the system purged until all hydrocarbons are eluted before introducing a new sample. In one study, this purging required about 2 hours (15).

The use of liquid air or nitrogen to cryogenically trap interfering species has been reported infrequently, but has been used successfully for samples containing 100 ppm CO or more (86,89). All interfering species except H_2 are trapped from the stream. Effectiveness for lower level CO samples has not been tested.

g) Conclusions

With careful attention to experimental procedure the iodine pentoxide method can provide precise and accurate results over a wide range of CO concentrations. Once the integrity of the system and activity of the I_2O_5 are confirmed by a blank corresponding to less than 1 ppm CO, it is an excellent referee method in the hands of a skilled analyst.

The need to maintain the I_2O_5 tube at operating temperature continuously makes this method difficult for use in the field.

.3 Oxidation with Mercuric Oxide

a) Description of the Method (4)

A 5ℓ sample is passed at a rate of 100 ml/min over red mercuric oxide heated to 200°C. Carbon monoxide is oxidized and Hg vapor is produced. The decrease in weight of the reaction tube due to loss of HgO is determined to the nearest 0.04 mg on a semimicrobalance.

b) Areas of Application

Ambient air.

c) Applicable Concentration Range

Studied over a range of 100-400 ppm, but should be applicable at higher levels.

d) Precision and Accuracy

Precision ranges from ±1 ppm at 100 ppm to ±1.4 ppm at 400 ppm. Accuracy is dependent on an accurate correction for the blank; accuracies of 1-2% were reported.

e) Interferences

All unsaturated hydrocarbons and higher saturated hydrocarbons react with HgO. Aromatics poison the HgO surface. The use of activated charcoal to remove these species is recommended. Hydrogen reacts to an extent of 50% of its true concentration at 200°C, but at 175°C only to the extent of 5%. Methane and water do not interfere.

f) Comments

This reaction is the basis for a trace CO analyzer for the 0.05-10 ppm range (17) which utilizes UV detection of liberated Hg.

This technique is applicable to continuous sampling, but a large sample volume is needed for adequate sensitivity when gravimetric analysis is used.

g) Conclusions

The method lacks sensitivity with gravimetric analysis and is not specific for CO. On this basis it is eliminated from further consideration.

.4.4 Miscellaneous Analytical Methods for CO

.1 Volumetric and Gravimetric Gas Analysis Methods

a) Description of Methods (22,38,39,51,56,81,82, 91,122,129,185)

A group of methods for determining high levels of CO are included in this section.

The volumetric methods are based on the measurement of the volume change resulting from the absorption of CO (or CO_2 after CO oxidation) by a liquid or solid reagent. Well known apparati such as the Orsat, Haldane, Bone and Wheeler, and Graham and Lawrence are based on this principle. After removing CO_2 in KOH, unsaturates in bromine water, and oxygen in pyrogallol, the carbon monoxide is absorbed in Cu_2Cl_2 (156,160, 208) or $CuSO_4$--β-naphthol solution (67,128), and the volume change is noted. A suspension of I_2O_5 in oleum has also been used (226). In an alternate approach, CO_2 and unsaturates are removed and the CO is oxidized to CO_2 over CuO heated to about 250°C (39,81,122), platinum at 600°C (38,54,56), or iodine pentoxide (129). The CO_2 is then removed in KOH solution, or cryogenically trapped at -180°C (39), and the reduction in volume is measured.

b) Areas of Application

Combustion gases and chemical process gases.

c) Applicable Concentration Range

The techniques are generally applicable over the range of 0.1 to 100%. The Orsat apparatus is limited to 0.3% (22), and the Haldane can detect a minimum of 0.02% (129).

d) Precision and Accuracy

Precision and accuracy of the Orsat apparatus is ±0.3% absolute; the Haldane, ±0.02% absolute. Precision and accuracy are primarily limited by volume measurement errors.

e) Interferences

Unremoved oxygen and unsaturates are absorbed along with CO by the liquid reagents.

f) Comments

Good technique is required to obtain precise results with volumetric gas analysis apparati.

g) Conclusions

The lack of sensitivity of these gravimetric and volumetric methods makes them inapplicable to the present study.

.2 Carbon Monoxide Indicating Tubes

a) Description of the Method (6,7,9,11)

A known volume of air is drawn through the indicator tube at about 100 ml/min. The tube contains silica gel impregnated with a reagent that undergoes a color change upon reaction with CO. The concentration of CO is determined by matching the color developed to standards. One of the best known indicator tubes uses a mixture of $PdSO_4$ and phosphomolybdic acid on silica. On exposure to CO the color changes from yellow to green and eventually to blue as the heteropoly acid is formed. Commercially available tubes are at best semi-quantitative, but by preparing the indicating gel fresh in the laboratory and using it the same day in conjunction with careful calibration, quantitative results can be obtained.

b) Areas of Application

Ambient air

c) Applicable Concentration Range

By varying the exposure time, CO from 2 ppb (20 min. exposure) to 4000 ppm (< 1 min. exposure) can be determined.

d) Precision and Accuracy

For the laboratory method, an accuracy of about 10% and reproducibility of ±5-10% were obtained in the 10-100 ppm

range (6).

e) Interferences

NO and NO_2 at levels of 3 ppm bleach the indicator color and invalidate results. A U-tube filled with $KMnO_4$ and ascarite reportedly eliminates the interference (9).

f) Comments

The steps required to impregnate the gel with reagent, dry it, and pack it into tubes is a lengthy process. Obtaining gels that work properly might require some practice.

g) Conclusions

The need to prepare fresh indicating gel daily makes the method too complex for further consideration. The NO, NO_2 levels (above) which bleach the color make this impractical for source measurements where NO_x concentrations are very high.

.3 Pyrotannic Acid Method

a) Description of the Method (45,131,153)

A 250 ml sample containing CO is shaken for 20 minutes with 2 ml of a dilute (20:1) solution of blood. About 0.04 g of pyrotannic acid (1:1 mixture of pyrogallic and tannic acids) is then added to the blood solution. After 30 minutes, the carmine color that develops in the presence of the CO-hemoglobin complex is compared to a standard color chart.

b) Areas of Application

Ambient air and CO in blood

c) Applicable Concentration Range

70-2000 ppm in presence of 21% O_2; if oxygen is reduced to 6%, 20 ppm can be determined.

d) Precision and Accuracy

A precision and accuracy of 50 to 300 ppm over the range 100-2000 ppm can be achieved. The accuracy depends greatly on calibration procedure and knowledge of O_2 concentration.

e) Interferences

HCN and H_2S must be removed.

f) Comments

Due to the equilibrium between the O_2 and CO complexes of hemoglobin, the O_2 partial pressure must be known for accurate results. Dried hemoglobin is commercially available (30), and should be more convenient to use than whole blood. The method however, has not been tested with that material.

g) Conclusions

The method as described is not sufficiently precise. The influence of O_2 is a fundamental limitation that cannot be easily overcome.

.4 Volumetric Analysis by Absorption in Blood (30,115)

a) Details of the Method

The CO in a 300 ml sample is complexed by shaking for 1 hour with 5 ml of hemoglobin solution. Prior to contact, the O_2 is removed from both the sample and the hemoglobin with $Na_2S_2O_4$. Dissolved gases are removed by shaking in vacuo. The bound CO is then liberated from the hemoglobin with potassium ferricyanide, and the volume change is measured in a Van Slyke apparatus.

b) Areas of Application

Ambient air.

c) Applicable Concentration Range

50-3000 ppm.

d) Precision and Accuracy

Recovery was reported to be 92 ±1.4% of theoretical. The precision at the 0.1% level was 1.3% (30).

e) Interferences

Highly soluble gases like acetylene may not be completely removed during the vacuum degassing prior to release of CO. Correction factors determined by using water instead of blood are recommended if soluble species are present (115). Possible interfering species that might bind to hemoglobin were not mentioned.

f) Comments

The method appears precise if carefully performed. Skill is obviously required to manipulate the 15 μℓ of CO in a 50 ppm sample. For accurate work with unknown contaminants a separate analysis using the "water blank" must be performed for each unknown.

g) Conclusions

The technique appears to be too complicated for further consideration.

5.0 Recommendations

The recommendations are divided into two groups: a) methods to meet immediate needs and b) promising methods which require further development.

The palladium chloride-bis(o-phenanthroline) and silver p-sulfamino benzoic acid methods are the preferred candidates for an immediate method since they have few interferences and are relatively simple. The latter has received more attention and should require less development effort.

Promising methods include the use of catalytic oxidation of CO with subsequent trapping in basic solution. This has potential as a continuous sampling method for CO from stacks provided CO_2 can be quantitatively removed with ascarite or another collection medium. Addition of organic bases in enzymes such as carbonic anhydrase or the alkaline medium should be studied since these are reported to increase the absorption efficiency for CO_2. The use of commercially available Hopcalite maintained at 100°C appears to be an improvement over iodine pentoxide as the catalyst. The use of Hopcalite or other mixed oxide catalysts at ambient temperature should be explored in more detail.

The intriguing possibility of developing a highly efficient, self-indicating catalyst for quantitative conversion of CO to CO_2 without CO_2 holdup at ambient temperatures should be considered. Such a catalyst would permit semi-quantitative estimation in the field at the same time a CO_2 sample is being obtained for more precise laboratory analysis. Scrubbing solutions such as ammonical cuprous chloride should be investigated for continuous collection of CO at low concentrations.

6.0 Summary and Conclusion

1. The iodine pentoxide method with iodometric finish is the best wet-chemical reference method for the laboratory. The simpler

Hopcalite catalytic oxidation should provide similar performance, especially if the range is limited to levels about 10 ppm. The efficiency of the Hopcalite is greater than the 97-99% of I_2O_5. Even if the I_2O_5 method is retained, determination of CO_2 formed would eliminate the interferences of H_2O and H_2. Much of the sensitivity of the I_2O_5 method to temperature changes is thought to result from spontaneous I_2O_5 decomposition; if CO_2 were measured this problem would be eliminated.

2. Oxidation by a catalyst is the only feasible alternative to grab sampling for subsequent laboratory analysis provided quantitative scrubbing of interfering flue components can be achieved.

3. The volumetric and gravimetric methods should be eliminated because they lack sensitivity and require a great deal of skill. However, collection in Cu+ scrubbing solution has promise as a result of the high solubility of CO.

4. Methods involving hemoglobin are not attractive since they require removal of O_2 from both the blood and the gaseous sample for reliable results.

5. The palladium chloride-bis(O-phenanthroline) and silver p-sulfaminobenzoic acid methods are very attractive because of their selectivity and simplicity . However, grab sampling techniques must be employed and the analysis time is long.

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